CHAPTER 2

NMR OIL TESTING EQUIPMENT

2.1 PURPOSE

To ensure that the FGIS sunflower oil program is applied uniformly by all specified service points, that only approved continuous wave or pulsed Nuclear Magnetic Resonance (NMR) equipment and procedures are used, that equipment maintenance and testing schedules are followed, and that laboratory setups conform to FGIS specifications.

2.2 APPROVED EQUIPMENT

The following equipment is approved for official NMR sunflower oil determinations.

Aluminum Dishes - Aluminum dishes with lids, 89 millimeters (mm) in diameter and 51 mm deep with closely fitted slip-on cover.

Balance - An approved electronic balance with 0.01 gram precision and RS-232 output (Mettler PM-200 or equivalent).

Bottle Brushes - Bottle brushes for cleaning NMR sample tubes.

Desiccating Cabinet - An airtight cabinet which can be equipped with a desiccant material to maintain a very low relative humidity environment.

Desiccant - Silica gel for drying, indicating, 6-16 mesh.

Electronic Power Conditioner (Optional) - An electronic power line conditioner (Sola model No. EPC 150-60, Tripp Lite model LS-604 or equivalent).

Forced Air Convection Oven - A forced air convection oven (Blue M Model OV-490A-2, or equivalent).

NMR Instruments - The Newport Analyzer Models Mark III-A and Oxford 4000 model (continuous wave wide-line NMR) equipped with type 10 magnet (Watson 10) and 150 milliliter (ml) magnet coil assembly. (Oxford Instruments Inc., 130-a Baker Avenue Extension, Concord, MA 01742, phone 1-800-447-4717).

Note: The Mark III-A model is approved for official use but is not in service at the current time. Therefore, the handbook does not contain step-by-step procedures for instrument operation.

Oxford Instruments pulsed NMR models MQA 6005 and MQA 7005 equipped with 5 Mhz magnet and 150 ml magnet coil assembly.

Universal Maran Ultra pulsed NMR equipped with 5 Mhz magnet and 150 ml magnet coil assembly. (Universal Systems, 29500 Aurora Road, Unit 16, Solon, OH 44139, phone 1-440-349-3210).

Bruker minispec mq7.5 pulsed NMR equipped with 7.5 Mhz magnet and 150 ml magnet coil assembly. (Bruker Canada Ltd., Milton, Ontario L9T 1Y6, Canada, phone 1-905-876-4641).

NMR Sample Tubes - NMR sample tubes for 150 mL NMR magnet coil assembly.

Rubber Stoppers - Rubber stopper for NMR sample tube, size No. 10.

Thermometers - Thermometer with 1EC to 51EC scale and 0.1EC divisions.

2.3 DISCLAIMER CLAUSE

The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned. Except for the NMR instrument, equivalent equipment may be used in place of the items listed.

2.4 OIL TESTING FACILITIES

Equipment location and environmental factors can affect the performance of NMR sunflower seed oil testing equipment. The space and facilities used by official personnel must meet the specifications outlined below.

a. Location of Equipment.

The method used for determining the oil content of sunflower seed samples utilizes a magnet with predetermined electromagnetic strength to activate the hydrogen atoms present in the sample. Metal objects or a strong magnet placed near the NMR equipment may interfere with the electromagnetic field and produce erroneous results. Therefore, do not place metal objects adjacent to the NMR equipment. **Do not place the NMR instrument on a steel table**.

A vibration-free table should be used to support equipment. Also, when more than one electronic instrument is located in the same work area, maintain at least 60 centimeters (approximately 2 feet) distance between instruments.

In addition, NMR equipment must be placed in a location conducive to a stable environment and shielded from electrical or electromagnetic interferences. NMR equipment must be protected from drafts, heating and cooling vents or devices, and preferably be kept away from outside walls and windows.

b. Temperature.

Maintain a constant temperature between 18EC (65EF) and 30EC (85EF) in the room where the NMR instrument is located. Record room temperature using a calibrated thermometer located near the NMR instrument.

Insert the thermometer into a small glass or plastic bottle filled with sunflower seed to reduce erroneous readings. The depth of the sunflower seed must be sufficient to cover the insertion level of the thermometer.

Fluctuation in room or sample temperature adversely affects analysis results. After calibration, a change in room or sample temperature greater than "0.5EC will require re-calibration using the SSS. Therefore, locate the NMR equipment in a room where the temperature remains very stable to minimize the need for re-calibration.

c. Power Supply.

The power for NMR instruments shall be supplied by a 120 " 10 VAC 15-20 amp dedicated circuit. A maximum of two electronic instruments and associated printers may be placed on one dedicated circuit (2 NMR or 1 NMR and 1 NIRT instrument). To reduce interference from other sources, do not place other equipment on the circuit.

d. Dust.

Accessible surfaces of the NMR instrument, balance, and surrounding area shall be maintained essentially free from contaminants. Use a vacuum cleaner and brush for maintaining a clean and dust-free environment in the sunflower seed testing area. **Do not** use compressed air for clean-up purposes.

At locations where a dust collection system is not available, place the NMR instrument and balance in a room separate from all dust-producing equipment such as grinders, dockage testers, and dividers.

2.5 INITIAL LABORATORY SETUP

Specified service points must observe certain guidelines when establishing new testing laboratories and/or placing new equipment in service.

a. New Laboratories.

Upon request, TSD will assist agencies in planning and preparing laboratories for official oil testing service. Agency managers must notify the field office manager that a new laboratory is being planned and provide a diagram of the proposed laboratory design. The diagram should contain the proposed locations of NMR oil testing equipment, location of major inspection equipment (e.g., dockage testers, dividers, etc.), and a description of the power supply. Any additional information regarding the laboratory setup or equipment should also be discussed.

The monitoring field office will forward a copy of all submitted information to the TSD for review. Upon receipt, the TSD will review the information and make recommendations to the agency and monitoring field office to facilitate the laboratory setup.

b. New Equipment.

Notify TSD before placing newly purchased NMR instruments in service. TSD will provide instructions to check the accuracy of the instrument and correct any deficiencies before the instrument is placed into official service. If problems are identified the checkout process may take several days to complete; therefore, contact TSD as soon as possible. Do not use newly purchased instruments for official NMR oil testing until the instrument has been checked and accepted by TSD.

NMR Calibration/Check Sample Log

Date of Initial Calibration: _____ (pulsed NMR instruments only)

Low SSS #: _____ Low SSS RV: _____ High SSS #: _____ High SSS RV: _____

Tolerance: If the measured value exceeds the reference value (RV) by +/- 0.3, retest the SSS. If the measured value still exceeds the RV by +/- 0.3 re-standardize the instrument and retest the SSS.

Date	Time	Temp.	Low SSS Value	Difference From RV	High SSS Value	Difference From RV	Temp.